Standard Operating Procedure for the Microwave Assisted Digestion Preparation of Solid Samples

1.0 Location

This procedure is performed in the spectroscopy laboratory, room 305.

2.0 Purpose

This method is used in the preparation of samples which require a more vigorous digestion process than the open hot plate (sec 9.32).

3.0 Scope

This procedure is used to prepare solid samples for metals analysis by atomic spectroscopy. It is more widely applicable to those sample matrices which are not acceptably digested by the use of the open hot plate method. It is not to be used for the digestion of oily wastes.

4.0 Reference

This method is referenced in CEM Microwave Sample Preparation System Applications Manual, NPDES, App. Note EN-3, Rev. Date 11-91.

5.0 Sample Handling and Preservation

Samples should be collected following the procedures for metals listed in the EPA document, Methods for Chemical Analysis of Water and Wastes, Rev. 3-83, Table 1.

6.0 Apparatus and Materials

6.1 CEM model MDS 2000 microwave oven.

6.2 Sample preparation apparatus including:

6.2.1 Teflon inserts and lids, acid washed and rinsed with deionized water.

6.2.2 Outer containers and lids.

6.2.3 Rupture membranes and tops.

6.2.4 Concentrated Nitric Acid, Instra-Analyzed--Trace Metal Free

7.0 Procedures

7.1 Weigh out a representative 0.5 g sub-sample into a teflon insert. Record the weight to the nearest 0.1 mg on printed spreadsheet. Do each sample in duplicate. One vessel, the pressure vessel, should contain a larger sample approximately 0.6000 g.

7.2 Place the insert into the outer marked container. Add 10 mL of conc. nitric acid to the insert.

7.3 Repeat steps 7.1 and 7.2 with even number of samples. Place vessels evenly spaced on turn table.

7.4 Prepare a duplicate and a spike as in 7.1 and 7.2. To the spike, include an additional 50 uL of 1000 ug/mL stock standard solution of the applicable metal. Do one blank and one LFB (lab fortified blank) spiked at the same level used for the matrix spike for each set of 10 samples

7.5 If samples, spikes, and duplicates total an odd number, include an additional blank.

7.6 Choose the sample which is most reactive (heaviest weight) and put the pressure control teflon lid on that sample. Use the cone shaped ferrule nut only for the armored temperature probe on the temperature control port. Place regular teflon lids on all remaining samples.

7.7 Cap vessels with outer lid. Place a rupture membrane into all teflon lids. Place the appropriate top into the teflon lid. Tighten the outer lids so that when turning the top the teflon lids do not turn.

7.8 Place turntable into microwave.

7.9 Flush the pressure control line by opening (position labeled open) the valve on the left side of the oven and pushing water through with the syringe. If the syringe is empty refill with deionized water which has been degassed with helium. Afterwards make sure that the valve to the pressure control line is closed (position labeled neutral).

7.10 Connect the pressure control line to the pressure monitoring outlet on the side of the pressure control teflon lid of the sample. The line should go through the center post and should not interfere with the circulating fan during table rotation. Make sure of this by rotating the table with only the pressure control vessel

hooked up. With the door open press F4 to start the turntable rotating to check that all lines are out of the way and turntable turns freely. Make sure door closes properly. Make sure line is completely filled or pressure sensor will not work. If pressure line does not seem to hold pressure, an O-ring can be inserted in the ferrule for the pressure line. For monitoring the temperature use the cone shaped ferrule nut only for the armored temperature probe. Insert glass thermowell and turn on cone shaped ferrule and then insert probe, making sure it is inserted completely and held firmly by ferrule. Make sure lines are free and do not interfere with the rotation of the turntable. Both pressure and temperature should be monitored in order to prevent high temperature damage (pinholes and over expanding) to vessels. When not using the temperature probe, close off temperature control port with a sealed ferrule nut.

7.11 Be sure to turn pressure valve to neutral or closed position for microwaving.

7.12 Recall the SEDIMENT method from memory. Verify that it looks like the following:

Stage	1	2	3	4	5
Power	75	75	75	75	75
PSI	50	90	150	180	190
Time	10:00	20:00	20:00	15:00	5:00
TAP	5:00	5:00	5:00	8:00	3:00
Temp	150	180	180	185	190
Fan	100	100	100	100	100

Additional information which is included in the method but does not affect the operating parameters is:

Vessels 6 Sample Weight 0.5g
Volume per Vessel 10mL Acid HNO3

Change power level when changing the number of vessels used according to the following: 8-12 vessels use 100%; 4 or less use 50%.

7.13 After making a final check including such things as the pressure control line valve closed, free rotation of the table, door properly closed, and temperature probe out of the way; begin the digestion by pressing the F4 button. Check that the pressure

rises and the turntable continues to turn properly. Press F2 to print run time data.

7.14 During the digestion determine the density of the sample in g/mL by weighing a representative portion to volume in a 10 mL volumetric flask. This will be used in the final calculations.

7.15 When method is completed, open and close door to stop rotation of the turntable. Allow the pressure to drop to between 30-60 psi before venting the pressure sample and removing the pressure line. Move entire turntable to hood and allow to cool with ventilation. Carefully vent each vessel. If the run proceeded satisfactorily--no rupture membranes broke--transfer the digested samples quantitatively to 100 mL volumetric flasks. Analyze the prepared samples with the instrument appropriate for that analyte.

8.0 Quality Assurance/Quality Control

Analyze at least one spike, duplicate and blank sample per batch.

9.0 Data Analysis

The result in ug/L should be calculated as follows:

Result = (A X B / C) X D X 1000 where

A = result from analysis in ug/L
B = Volume to which the digested samples were brought in L (0.100 L)
C = Weight of the sample digested in g
D = Density of the sample in g/mL

10.0 Documentation

Record all weights and keep all records of analysis. Enter the calculated value into the lims via a worklist. Keep all other relevant information with the worklist and place into the individual metal's parameter log book.

11.0 Records

All recorded information shall be maintained and kept in the relevant metal's log books.